

Bis(2-hydroxyiminomethyl-6-methoxyphenolato- κ^2N,O^1)copper(II)

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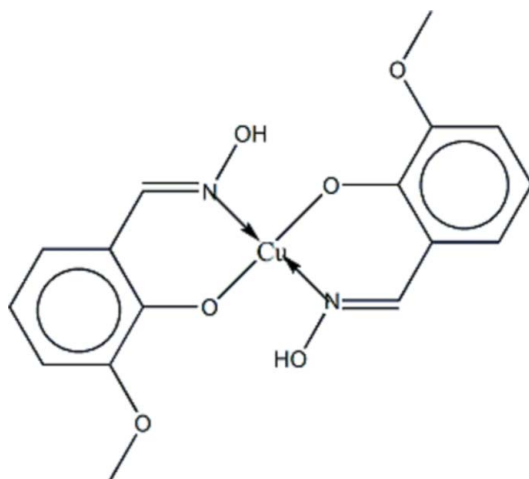
Received 22 June 2012; accepted 15 July 2012

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(C-C) = 0.002$ Å; R factor = 0.028; wR factor = 0.077; data-to-parameter ratio = 17.0.

In the title compound, $[Cu(C_8H_8NO_3)_2]$, the nearly planar molecule (r.m.s. deviation = 0.037 Å) is centrosymmetric with the Cu^{II} atom lying on an inversion center. The Cu^{II} atom is tetracoordinated, displaying a slightly distorted square-planar geometry. The main deviation from the ideal geometry is seen in the differences in the $Cu-O$ [1.8833 (10) Å] and $Cu-N$ [1.9405 (13) Å] bond lengths, while angular deviations are less than 3° . Intramolecular $O-H \cdots O$ and intermolecular $Csp^2-H \cdots O$ hydrogen bonds form $S(5)$ and $R_2^2(8)$ ring motifs, respectively. The latter interaction results in chains of molecules along [100].

Related literature

For related structures, see: Zhang *et al.* (2008); Li *et al.* (2004), 2009). For bond-valence-sum calculations, see: Brown & Altermatt (1985). For *in situ* formation of polydentate ligands, see: Coxall *et al.* (2000). For background to direct synthesis, see: Makhankova (2011).



Experimental

Crystal data

$[Cu(C_8H_8NO_3)_2]$
 $M_r = 395.85$
Monoclinic, $P2_1/n$
 $a = 8.4906$ (4) Å
 $b = 4.8997$ (2) Å
 $c = 18.9309$ (9) Å
 $\beta = 94.906$ (4) $^\circ$
 $V = 784.67$ (6) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 1.43$ mm⁻¹
 $T = 293$ K
 $0.50 \times 0.20 \times 0.10$ mm

Data collection

Oxford Diffraction Xcalibur/
Sapphire3 diffractometer
Absorption correction: multi-scan
(*CrysAlis RED*; Oxford
Diffraction, 2010)
 $T_{min} = 0.535$, $T_{max} = 0.763$
8497 measured reflections
2245 independent reflections
1816 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.077$
 $S = 1.01$
2245 reflections
132 parameters
H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{max} = 0.39$ e Å⁻³
 $\Delta\rho_{min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, $^\circ$).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|---------------------|------------|--------------|--------------|----------------|
| $O3-H3O \cdots O1$ | 0.82 | 1.94 | 2.5840 (16) | 134 |
| $C7-H7 \cdots O3^i$ | 0.903 (19) | 2.49 (2) | 3.3231 (19) | 154.3 (15) |

Symmetry code: (i) $x + 1, y, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2010); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LR2071).

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supplementary materials

Acta Cryst. (2012). E68, m1165 [doi:10.1107/S1600536812032187]

Bis(2-hydroxyiminomethyl-6-methoxyphenolato- κ^2N,O^1)copper(II)

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Comment

Aiming to prepare Cu/Mn heterometallic complexes with an ONO donor Schiff base (H_2L = 2-hydroxyiminomethyl-6-methoxyphenol) the following system based on "direct synthesis" methodology (Makhankova, 2011) has been investigated: $Cu^0 - Mn^0 - o$ -vianillin– $NH_2OH \cdot HCl - NH_4X - solv$ (in open air), where o -vianillin = 2-hydroxy-3-methoxybenzaldehyde; $X = Cl, Br, I$; $solv = CH_3OH, dimethylformamide(dmf), dimethylsulfoxide(dmsO)$.

In all cases the total dissolution of copper and manganese powders was observed within 5 - 6 h resulting into intensive dark green solutions. X-ray quality crystals were obtained from the systems with NH_4Br in $dmsO$ and NH_4Cl in dmf , but in the former one the yield was some better.

The asymmetric unit of $[Cu(HL)_2]$ includes one-half of the molecule with Cu atom occupying the (1/2 1/2 1/2) special position of multiplicity 2. The coordination geometry of the metal atom is square-planar, with the CuN_2O_2 chromophore, formed by means of two imine nitrogen atoms and two phenolate oxygen atoms of the two monodeprotonated Schiff base ligands realising their bidentate chelate function, [1.1,1,0] by Harris notation (Coxall *et al.*, 2000) (Fig. 1). Difference between Cu–O and Cu–N bond lengths (Table 1) causes significant linear distortion of the square. Deviations in bond angles at the Cu atom are less than 3° . The bond valence sum analysis applied to the appropriate bond lengths supports the +2 oxidation state for copper, $BVS(Cu) = 2.003$ (Brown & Altermatt, 1985).

Hydrogen bonds (HBs) play the principal role in the crystal structure of $[Cu(HL)_2]$. The N–OH group takes part in simultaneous formation of a strong intramolecular $O(3)–H(3O) \cdots O(1)$ hydrogen bond with $O_{phenolate}$ of the second ligand and a weak *inter*-molecular $C(7)–H(7) \cdots O(3')$ hydrogen bond with $C(sp^2)–H$ group of the neighboring molecule (Fig. 2, Table 2). As a result, two ligands being coordinated to the Cu^{II} ion form some analogue of a macrocyclic ligand [R14] based on HBs which binds to the copper center generating two 6-membered (with only covalent bonds) and two 5-membered (with covalent and hydrogen bonds) rings (Fig. 1). It is worth noting that all known structures with H_2L , namely $Co(HL)_2$ (Zhang *et al.*, 2008), $Ni(HL)_2$ (Li *et al.*, 2009) and $VO(HL)_2$ (Li *et al.*, 2004), are built in the same manner demonstrating high thermodynamic stability of such structure.

The adjacent molecules join through complementary $C–H \cdots O$ HBs, $[R_2^2(8)]$ synthon, forming one-dimensional stair-like ribbons along (100) direction (Fig. 2).

Experimental

Copper powder (0.06 g, 1 mmol), manganese powder (0.05 g, 1 mmol), o -vianillin (0.46 g, 3 mmol), hydroxylamine hydrochloride (0.21 g, mmol) and NH_4Br (0.20 g, 2 mmol) were added to 10 ml of dimethylsulfoxide. The mixture was stirred magnetically at 323 – 333 K until total dissolution of metal powders was observed (*ca* 5 h). Goldish-green needle crystals that precipitated after 1 day, were collected by filtration, washed with methanol and dried in air; yield 32% based on Cu. IR(KBr, cm^{-1}): 3080(*m*), 3057(*m*), 3006(*m*), 2959(*m*), 2936(*m*), 2834(*m*), 1649(*m*), 1598(*m*), 1554(*w*), 1510(*m*), 1468(*s*), 1451(*s*), 1353(*w*), 1332(*m*), 1302(*s*), 1247(*s*), 1216(*s*), 1195(*m*), 1101(*m*), 1081(*m*), 1018(*m*), 969(*s*), 932(*w*),

863(*m*), 778(*m*), 755(*m*), 735(*s*), 712(*s*), 624(*m*), 575(*w*), 546(*w*), 502(*w*).

Refinement

Structure was solved by direct method and refined against F^2 within anisotropic approximation for all non-hydrogen atoms. All hydrogen atoms were located from difference Fourier map and refined isotropically, except phenyl (H(3) - H(5)) and hydroxyl (H(3O)) H atoms that were allowed to ride on their attached atoms with $C-H = 0.93$ (1) Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for phenyl, and $C-H = 0.82$ (1) Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for hydroxyl H atoms. Coordinates of Cu(1) were constrained to special position ($x=0.5000$, $y=0.5000$, $z=0.5000$).

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2010); cell refinement: *CrysAlis CCD* (Oxford Diffraction, 2010); data reduction: *CrysAlis RED* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

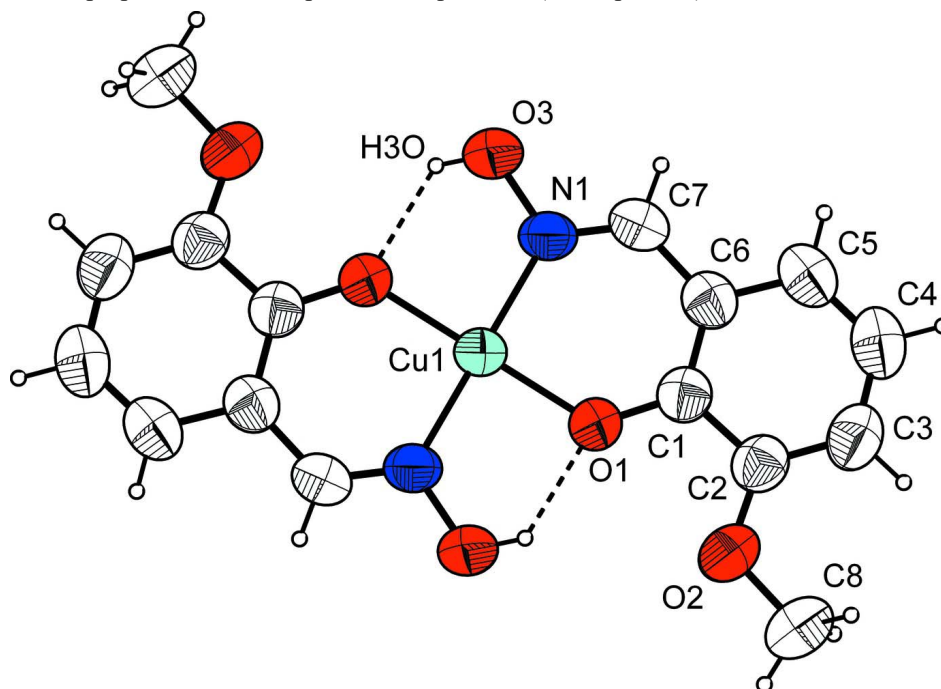
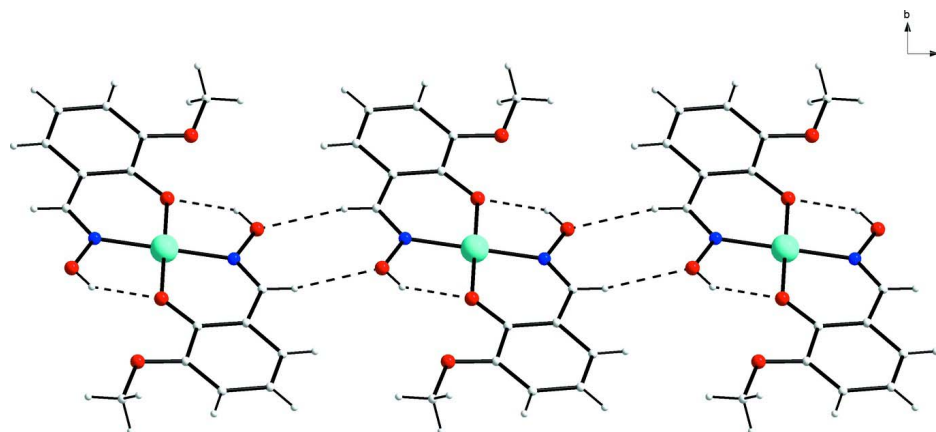


Figure 1

Molecular structure of $[Cu(HL)_2]$ with 70% probability displacement ellipsoids for non-H atoms. The dashed lines denote hydrogen bonds.

**Figure 2**

Fragment of chain-like alignment of $[\text{Cu}(\text{HL})_2]$ viewed along the $[100]$ direction.

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Crystal data

$[\text{Cu}(\text{C}_8\text{H}_8\text{NO}_3)_2]$

$M_r = 395.85$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 8.4906\ (4)\ \text{\AA}$

$b = 4.8997\ (2)\ \text{\AA}$

$c = 18.9309\ (9)\ \text{\AA}$

$\beta = 94.906\ (4)^\circ$

$V = 784.67\ (6)\ \text{\AA}^3$

$Z = 2$

$F(000) = 406$

$D_x = 1.675\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2721 reflections

$\theta = 3.1\text{--}32.2^\circ$

$\mu = 1.43\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Needle, gold-green

$0.50 \times 0.20 \times 0.10\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur/Sapphire3
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: $16.1827\ \text{pixels mm}^{-1}$

ω scans

Absorption correction: multi-scan

(*CrysAlis RED*; Oxford Diffraction, 2010)

$T_{\min} = 0.535$, $T_{\max} = 0.763$

8497 measured reflections

2245 independent reflections

1816 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 3.9^\circ$

$h = -11 \rightarrow 11$

$k = -6 \rightarrow 6$

$l = -26 \rightarrow 26$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.077$

$S = 1.01$

2245 reflections

132 parameters

0 restraints

11 constraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.39\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.19\ \text{e \AA}^{-3}$

Special details

Experimental. CrysAlis RED, Oxford Diffraction Ltd., 2010. Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|--------------|----------------------------------|
| Cu1 | 0.5000 | 0.5000 | 0.5000 | 0.03633 (10) |
| N1 | 0.27576 (15) | 0.5566 (3) | 0.50728 (7) | 0.0398 (3) |
| C1 | 0.60680 (18) | 0.0698 (3) | 0.59715 (8) | 0.0378 (3) |
| O1 | 0.49066 (12) | 0.2223 (2) | 0.56829 (5) | 0.0446 (2) |
| O2 | 0.41409 (14) | −0.1275 (2) | 0.66227 (6) | 0.0541 (3) |
| C2 | 0.56941 (19) | −0.1252 (3) | 0.64921 (7) | 0.0416 (3) |
| O3 | 0.20159 (13) | 0.3924 (3) | 0.55453 (6) | 0.0518 (3) |
| H3O | 0.2665 | 0.2879 | 0.5745 | 0.078* |
| C3 | 0.6860 (2) | −0.2899 (3) | 0.68168 (8) | 0.0488 (4) |
| H3 | 0.6603 | −0.4164 | 0.7155 | 0.059* |
| C4 | 0.8415 (2) | −0.2687 (3) | 0.66435 (8) | 0.0512 (4) |
| H4 | 0.9189 | −0.3814 | 0.6865 | 0.061* |
| C5 | 0.8809 (2) | −0.0838 (4) | 0.61520 (9) | 0.0467 (3) |
| H5 | 0.9853 | −0.0700 | 0.6043 | 0.056* |
| C6 | 0.76430 (18) | 0.0881 (3) | 0.58031 (8) | 0.0393 (3) |
| C7 | 0.81554 (18) | 0.2753 (3) | 0.52815 (8) | 0.0420 (3) |
| H7 | 0.920 (2) | 0.279 (4) | 0.5214 (9) | 0.054 (5)* |
| C8 | 0.3647 (3) | −0.3339 (4) | 0.70831 (10) | 0.0572 (4) |
| H8A | 0.392 (3) | −0.514 (4) | 0.6925 (12) | 0.050 (6)* |
| H8B | 0.418 (3) | −0.314 (4) | 0.7595 (12) | 0.072 (6)* |
| H8C | 0.255 (3) | −0.322 (4) | 0.7053 (10) | 0.064 (6)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|--------------|-------------|-------------|
| Cu1 | 0.03102 (14) | 0.03680 (14) | 0.04131 (15) | −0.00245 (9) | 0.00388 (9) | 0.00387 (9) |
| N1 | 0.0332 (6) | 0.0452 (6) | 0.0413 (6) | −0.0056 (5) | 0.0060 (5) | 0.0017 (5) |
| C1 | 0.0405 (7) | 0.0349 (6) | 0.0379 (7) | −0.0014 (6) | 0.0017 (6) | −0.0020 (5) |
| O1 | 0.0358 (5) | 0.0457 (5) | 0.0525 (5) | 0.0010 (4) | 0.0052 (4) | 0.0134 (5) |
| O2 | 0.0521 (7) | 0.0494 (6) | 0.0621 (7) | −0.0034 (5) | 0.0118 (5) | 0.0163 (6) |
| C2 | 0.0463 (8) | 0.0360 (7) | 0.0422 (7) | −0.0041 (6) | 0.0021 (6) | −0.0008 (6) |
| O3 | 0.0370 (5) | 0.0640 (7) | 0.0550 (6) | −0.0058 (6) | 0.0083 (5) | 0.0176 (6) |
| C3 | 0.0611 (10) | 0.0393 (7) | 0.0451 (7) | −0.0020 (7) | −0.0013 (7) | 0.0044 (6) |
| C4 | 0.0554 (9) | 0.0464 (8) | 0.0499 (8) | 0.0099 (7) | −0.0072 (7) | −0.0004 (7) |
| C5 | 0.0402 (8) | 0.0483 (7) | 0.0505 (8) | 0.0060 (7) | −0.0024 (7) | −0.0055 (7) |

| | | | | | | |
|----|-------------|------------|-------------|-------------|-------------|-------------|
| C6 | 0.0391 (7) | 0.0369 (6) | 0.0410 (7) | 0.0000 (6) | −0.0009 (6) | −0.0049 (6) |
| C7 | 0.0320 (7) | 0.0477 (8) | 0.0463 (7) | −0.0020 (6) | 0.0033 (6) | −0.0023 (6) |
| C8 | 0.0656 (12) | 0.0517 (9) | 0.0560 (10) | −0.0083 (9) | 0.0151 (9) | 0.0089 (8) |

Geometric parameters (Å, °)

| | | | |
|---|--------------|------------------------|--------------|
| Cu1—O1 ⁱ | 1.8833 (10) | C3—C4 | 1.392 (3) |
| Cu1—O1 | 1.8833 (10) | C3—H3 | 0.9300 |
| Cu1—N1 | 1.9405 (13) | C4—C5 | 1.361 (2) |
| Cu1—N1 ⁱ | 1.9405 (13) | C4—H4 | 0.9300 |
| N1—C7 ⁱ | 1.281 (2) | C5—C6 | 1.419 (2) |
| N1—O3 | 1.3928 (17) | C5—H5 | 0.9300 |
| C1—O1 | 1.3179 (17) | C6—C7 | 1.442 (2) |
| C1—C6 | 1.404 (2) | C7—N1 ⁱ | 1.281 (2) |
| C1—C2 | 1.428 (2) | C7—H7 | 0.903 (19) |
| O2—C2 | 1.362 (2) | C8—H8A | 0.965 (18) |
| O2—C8 | 1.422 (2) | C8—H8B | 1.04 (2) |
| C2—C3 | 1.380 (2) | C8—H8C | 0.93 (2) |
| O3—H3O | 0.8200 | | |
| O1 ⁱ —Cu1—O1 | 180.0 | C4—C3—H3 | 119.7 |
| O1 ⁱ —Cu1—N1 | 92.56 (5) | C5—C4—C3 | 120.27 (15) |
| O1—Cu1—N1 | 87.44 (5) | C5—C4—H4 | 119.9 |
| O1 ⁱ —Cu1—N1 ⁱ | 87.44 (5) | C3—C4—H4 | 119.9 |
| O1—Cu1—N1 ⁱ | 92.56 (5) | C4—C5—C6 | 120.76 (16) |
| N1—Cu1—N1 ⁱ | 180.00 (8) | C4—C5—H5 | 119.6 |
| C7 ⁱ —N1—O3 | 114.91 (13) | C6—C5—H5 | 119.6 |
| C7 ⁱ —N1—Cu1 | 127.46 (11) | C1—C6—C5 | 119.77 (15) |
| O3—N1—Cu1 | 117.60 (10) | C1—C6—C7 | 123.03 (14) |
| O1—C1—C6 | 124.28 (14) | C5—C6—C7 | 117.20 (15) |
| O1—C1—C2 | 117.57 (14) | N1 ⁱ —C7—C6 | 124.28 (14) |
| C6—C1—C2 | 118.15 (14) | N1 ⁱ —C7—H7 | 117.8 (12) |
| C1—O1—Cu1 | 128.33 (10) | C6—C7—H7 | 117.8 (12) |
| C2—O2—C8 | 117.25 (14) | O2—C8—H8A | 111.8 (14) |
| O2—C2—C3 | 125.65 (14) | O2—C8—H8B | 112.2 (12) |
| O2—C2—C1 | 113.99 (13) | H8A—C8—H8B | 106.2 (18) |
| C3—C2—C1 | 120.35 (15) | O2—C8—H8C | 105.2 (13) |
| N1—O3—H3O | 109.5 | H8A—C8—H8C | 107.6 (19) |
| C2—C3—C4 | 120.69 (15) | H8B—C8—H8C | 113.9 (17) |
| C2—C3—H3 | 119.7 | | |
| O1 ⁱ —Cu1—N1—C7 ⁱ | 2.42 (14) | C6—C1—C2—C3 | 0.0 (2) |
| O1—Cu1—N1—C7 ⁱ | −177.58 (14) | O2—C2—C3—C4 | −179.65 (14) |
| O1 ⁱ —Cu1—N1—O3 | −179.73 (11) | C1—C2—C3—C4 | 0.1 (2) |
| O1—Cu1—N1—O3 | 0.27 (11) | C2—C3—C4—C5 | 0.3 (2) |
| C6—C1—O1—Cu1 | −0.8 (2) | C3—C4—C5—C6 | −0.6 (2) |
| C2—C1—O1—Cu1 | 178.82 (10) | O1—C1—C6—C5 | 179.24 (14) |
| N1—Cu1—O1—C1 | −177.96 (12) | C2—C1—C6—C5 | −0.4 (2) |
| N1 ⁱ —Cu1—O1—C1 | 2.04 (12) | O1—C1—C6—C7 | −1.0 (2) |
| C8—O2—C2—C3 | −6.2 (2) | C2—C1—C6—C7 | 179.38 (13) |

| | | | |
|-------------|--------------|--------------------------|--------------|
| C8—O2—C2—C1 | 174.03 (14) | C4—C5—C6—C1 | 0.7 (2) |
| O1—C1—C2—O2 | 0.10 (19) | C4—C5—C6—C7 | −179.06 (14) |
| C6—C1—C2—O2 | 179.74 (13) | C1—C6—C7—N1 ⁱ | 0.6 (2) |
| O1—C1—C2—C3 | −179.64 (13) | C5—C6—C7—N1 ⁱ | −179.65 (15) |

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|---------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O3—H3O \cdots O1 | 0.82 | 1.94 | 2.5840 (16) | 134 |
| C7—H7 \cdots O3 ⁱⁱ | 0.903 (19) | 2.49 (2) | 3.3231 (19) | 154.3 (15) |

Symmetry code: (ii) $x+1, y, z$.